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A Method for Out-of-autoclave Fabrication of High Fiber Volume Fraction Fiber Reinforced Polymer Composites

by Larry R. Holmes, Jr., James P. Wolbert, and Jared M. Gardner

ARL-TR-6057 **July 2012**

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14. ABSTRACT
The U.S. Army Research Laboratory (ARL) has a long history in the fabrication of composite materials for both lightweight structures and vehicular armor. Through the years, ARL research has focused on cost-efficient materials and processes with Army-specific goals in mind. While retaining the Army perspective, we are focusing on basic scientific fundamentals that lead to high quality, cost-effective composite systems for uses across the scientific spectrum. In our research, we have been increasing the fiber-volume fraction by vacuum-assisted resin transfer molding (VARTM) in order to produce composite structures with aerospace-grade qualities. Of specific focus is the control of processing parameters during resin infusion to obtain fiber-volume fractions like those of autoclave-processed composites. Using a combination of viscosity control, ARL-based VARTM techniques, and a pressure control system, we increased the fiber-volume content from 50% (ARL's normal processing range for a particular material system and VARTM process) to over 60%. Future work will focus on achieving a 65% fiber-volume fraction with this material system. This jump in fiber-volume fraction can provide higher strength-to-weight ratios in composite parts while cutting the fabrication cost. As a result, we are well on the way to providing cost-effective advanced composite materials. Processing characteristics are presented and evaluations are discussed.

15. SUBJECT TERMS
Out-of-autoclave, fiber volume, composite processing, composite fabrication

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1. Introduction

Composite materials can be defined as a combination of two or more materials with distinct material properties that, when combined, create a new material with properties not attainable by any of the individual constituent materials. These engineered material systems can have rigidity, strength, weight, density, impact resistance, electrical and optical properties, and many other material properties that are manipulated to suit a desired function. Traditional fiber-filled composites are currently used as multifunctional materials. Conductive fibers can be imbedded into a polymer matrix providing a fully insulated electrical system, like those used in computer chips. Fiber mat composites can provide strength as well as a thermal insulating barrier, like the ones used in aircraft housings. These and many more multifunctional composite materials obtain their respective multifunctional properties by controlling the fibers or particles dispersed into the matrix material (*1*). This effort examines a novel processing technique in the fabrication of continuous fiber polymer composite systems that are designed for optimal strength, conventionally typified by high fiber-volume fraction (fvf). We show that a combination of resin viscosity control and proper compaction and fabric nesting can produce high fvf composites out-of-autoclave while saving time and reducing cost.

Traditionally, fabrication of composites via resin infusion has been associated with low fabrication repeatability and a lack in dimensional tolerances versus prepreg composites fabricated in an autoclave. However, recent advancements in process understanding have allowed for the fabrication of structural, aerospace-grade composites via out-of-autoclave processing. Examples of vacuum-assisted resin transfer molding (VARTM) processed technology demonstrators include the C-17 main landing gear door and forward pylon of the Chinook, which have met performance requirements for military components; and multiple primary structural components for civil air transportation, such as the Airbus A380 flap tracks, Boeing 787 pressure bulk head, and Mitsubishi Heavy Industries (MHI) vertical stabilizer for the Mitsubishi Regional Jet (MRJ), all of which are currently in production (*2*–*9*).

A goal of this endeavor is the development and evaluation of a processing method for the fabrication of high fvf, thick cross-sectional composites at a relatively low cost. Much of a composite's material and mechanical properties are related to the fvf of the structure, and while high fvf composite laminates are attainable in autoclave processing, these techniques may not be cost effective (*10*–*15*). The out-of-autoclave process described in this work has the potential to reduce fabrication costs while still providing for high fvf composite laminates. The concept of out-of-autoclave processing of composites is not novel, and there has been much effort in this area, the most common of these approaches being the Seeman Composite Resin Infusion Molding Process (SCRIMP), the controlled atmospheric pressure resin infusion (CAPRI) process, and the vacuum-assisted process (VAP) (*16*–*18*). There is much documentation on the

comparison of these process and others, which is out of the scope of this effort (*2, 19–21*). However, some characteristics associated with these processes are built upon, including material selection, process description, and results of fvf analysis.

2. Experimentation

2.1 Materials

2.1.1 Fabric Selection

In this investigation, a material system was selected based on opportunity for process control and improvement. The fvf of composite structures fabricated using resin infusion methods also vary widely due to multiple materials related variables, including fiber diameter, fiber sizing, yarn shape, yarn twist, weave pattern, ply stack orientation, resin viscosity, resin compatibility, and countless others. The fabric chosen for this study is a 831 g/m^2 240 style 5 x 5 plain weave, S-2 fiberglass (BGF, Greensboro, NC). In composite fabrication, compaction of the preform is related to the nesting of fibers, both intra- and inter-laminarly. Of the systems common to U.S. Army Research Laboratory (ARL) operations, a heavy plain weave, like this one, is the most difficult to nest and therefore it becomes very difficult to obtain high fvfs. As seen in figure 1, the plain weave is characterized by its balance, meaning it has two orthogonal yarns with a nominally equal number of tows.

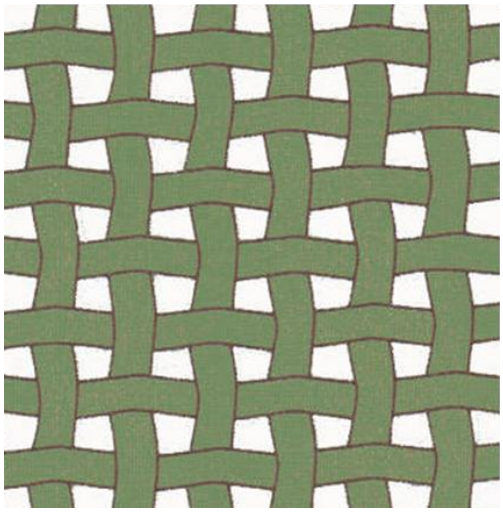

Figure 1. Image of a plain fabric weave courtesy of Pamela Cole Harris's *Knowing Your Fabric Weave – Basic and Plain Weave* (*22*).

The plain weave is also balanced in the crimp of the tows, which is the "waviness of the fiber" expressed numerically as the "number of crimps (waves) per unit length." This means that the warp and weft tows interlace alternately one over the other (*23*). While drape and in-plane

permeability normally increase with an increase in crimp (*23*), the laminate compaction, and eventually fvf, may suffer. With these characteristics in mind, this fabric was selected as a "worst case" specimen for our operation. Characteristics of this fabric can be seen in table 1 from the manufacturer's technical data sheet (*24*).

Table 1. Manufacturer's data sheet for selected fabric (*24*).

Style	Finish	Weave Pattern	Yarn in Warp	Yarn in Weft	Count Ends x Picks	Weight g/m^2 (oz/yd^2)	Breaking Strength Warp kN/cm (lb/in)	Breaking Strength Weft kN/cm (lb/in)	Thickness m (in)
240	463 Epoxy Compatible	Plain	463-AA-250 Yield S Glass Roving	463-AA-250 Yield S Glass Roving	5 x 5	813 (24.00)	1.313 (750)	1.366 (780)	0.008 (0.300)

2.1.2 Resin Selection

Many of the composites fabricated by resin infusion methods at ARL are made with the legacy SC-15 epoxy (Applied Poleramic Inc., Benicia, CA) resin system. SC-15 is a low viscosity, two-phase toughened epoxy system that has been specifically designed for resin infusion processes. An image of a contact angel experiment of Part A of SC-15 can be seen in figure 2 (*25*).

Figure 2. Image of contact angle experiment of SC-15 Part A. Image courtesy of reference 25.

There is a significant amount of detailed documentation regarding the SC-15 epoxy system and vast experience in processing of composites with it (*25*–*33*). For these reasons, the SC-15 system was selected. Characteristics of this resin system can be seen in table 2 and are courtesy of the manufacture's data sheet and experimental data.

Table 2. Technical data for SC-15 epoxy resin system (*25*, *33*).

System	SC-15 Toughened Two Phase, Two Part Epoxy
Mix Ratio of A:B (wt)	100:30
Viscosity (cP) at ambient	350
Application Temp °C (°F)	25 (77)
Time to Reach 700 cP at Application Temperature (hr)	3.25
Suggested Cure Cycle (h)	12 at 25 °C (77 °F)
Suggested Post Cure (h)	2 at 93.3 °C (200 °F)
Water pick up (%)	1.3
Density * (kg/m^2)	1198
Surface Tension * N/m	$3.7x10^{-2}$ ($\pm 2x10^{-4}$)

* Denotes data from reference 25, all other data from reference 33.

2.2 Processing

The fiberglass-reinforced composite plates used as the control samples in these experiments were fabricated using the VARTM process, resulting in distinct tool side and bag side surfaces which differed in flatness and roughness; for a complete overview of the VARTM process see reference 34. Approximately 95 kPa (14 psi) was achieved in the vacuum bag during processing. A vacuum drop test was performed for 1 min using a pressure gauge in line with the vacuum source, demonstrating no greater than 10 kPa (1.5 psi) pressure change in the vacuum bag. A redundant second vacuum bag was applied over the entire layup to further protect against leaks and decrease potential process variability across the sample set. This double bag VARTM is the standard practice at ARL; for this reason, it was used as the control in this investigation. The dry preform is then held under vacuum for 12 h for debulk. All fiberglass laminates were fabricated using the SC-15 low viscosity epoxy resin system, as described in the materials section of this document. The VARTM process is illustrated in figures 3 and 4 (*35*).

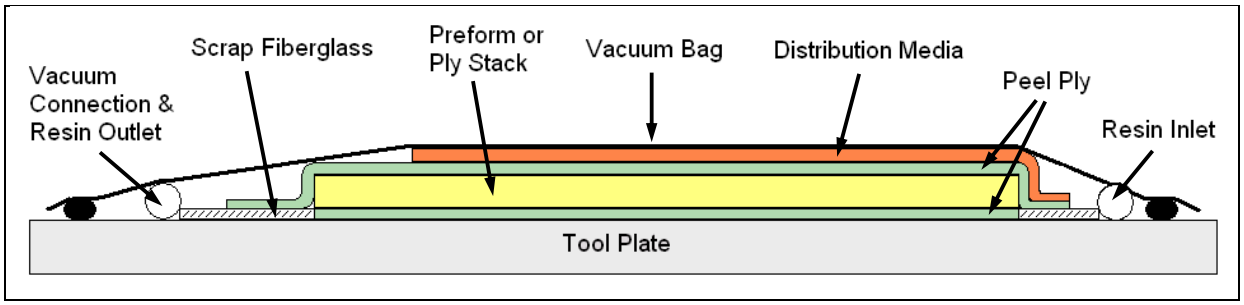

Figure 3. Schematic showing the side view of a composite plate being fabricated by the VARTM process with a single bag.

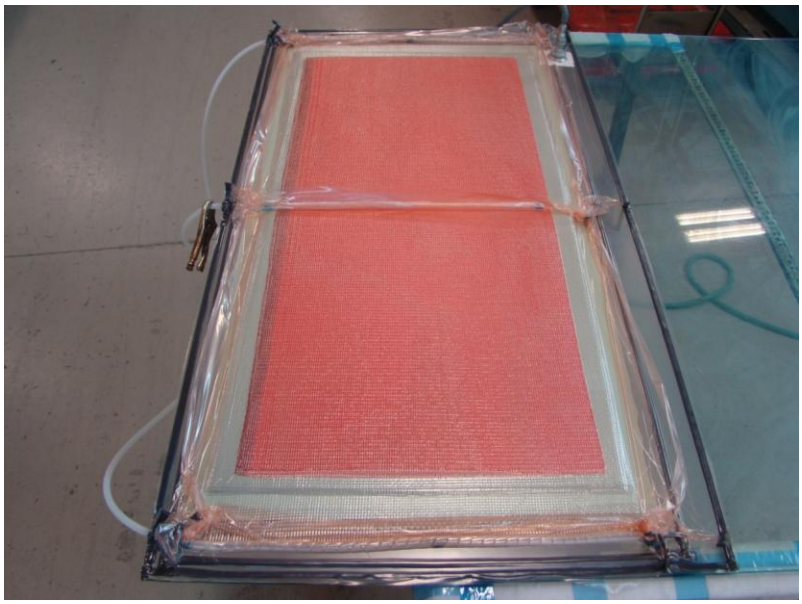

Figure 4. Image of fiberglass composite being fabricated using VARTM processing.

Experimentation in literature has shown that better preform compaction can be achieved via multiple debulk cycles, specifically, research has shown an optimal compaction of a 15-ply E-glass preform to be obtained after approximately 350 debulking cycles (figure 5a). This large number of debulk cycles is not an efficient manner of processing as this would drastically increase fabrication time. It is also noted that the permeability of the dry preform under debulking and compaction can decrease significantly, in some instances, by a factor of 10 (figure 5b) (*36*).

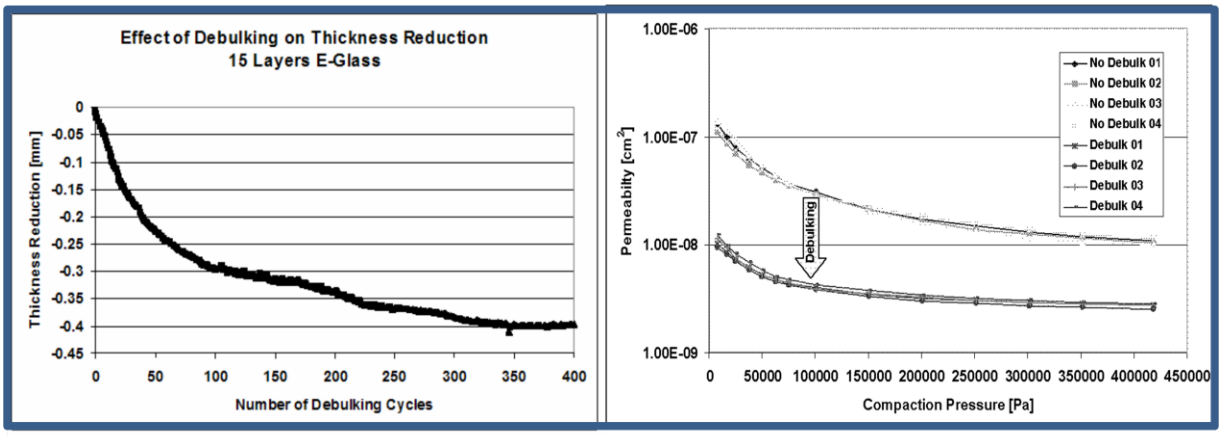

Figure 5. (a) (Left) Results showing optimal compaction of an E-glass (similar compaction to S-Glass) laminate at approximately 350 debulk cycles and (b) (right) decrease in permeability due to compaction of fabric preform (*36*).

This decrease in permeability of the preform can result in much higher impregnation times as it becomes more difficult to move the resin through the compacted preform. This theory was proven through experimentation described later, but a comprehensive study of the permeability of a compacted preform can be seen in reference 37. In an effort to solve the issues of increased fabrication times associated with multiple debulk cycles, a computer-controlled hydraulic press, shown in figure 6, was used to decrease compaction time compared to the 350 debulk cycles.

It was determined that less than 241 kPa (35 psi) compaction in a hydraulic press could be used to obtain a nesting of the preform like that associated with the previous referenced study (*36*). It should be noted that a computer-controlled press was used here in order to be sure of repeatability and control in this academic experiment; however, 241 kPa (35 psi) could easily obtained through other methods in a manufacturing scale, which may include clamping techniques, a manual press, or simply a heavily weighted topside tool.

Figure 6. Image of a thermally controlled hydraulic press.

3. Results

Multiple 61 cm x 61 cm (24 in x 24 in) 24-ply composite laminates with a cross-sectional thickness of approximately 1.3 cm (0.5 in) were fabricated using a computer-controlled press to obtain a high compaction pressure on the preform. The preform, nested at 241 kPa (35 psi), was infused while the press maintained this constant pressure. All samples were tested according to ASTM D-2584, a standard for determining fiber content in composite laminates (*38*). A summary of the results can be seen in table 3 at the end of this section. The control samples fabricated using the double bag VARTM technique had the following constituents as determined by the standard:

Average Fiber Volume = 50.1%

Average Resin Volume = 49.0%

Average Void Volume = 0.9%

Average Infusion Time = 30 min

These results are typical of our current double bag VARTM technique with this material system, which allows for near 50/50 fiber to resin ratio with low void content.

Table 3. Summary of results.

Processing Parameters	Average Fiber Volume (%)	Average Resin Volume (%)	Average Void Volume (%)	Average Infusion Time (min)
1. Double bag VARTM control	50.07	49.00	0.90	30
2. Compaction only 241 kPa (35 psi)	52.38	46.23	1.40	56
3. Heated preform 48.9 °C (120 °F) and compaction 241 kPa (35 psi)	56.56	42.50	0.95	49
4. Heated resin 48.9 °C (120 °F), heated preform 48.9 °C (120 °F), and compaction 241 kPa (35 psi)	60.39	38.37	1.25	32

The first experimental process with compaction and infusion in the hydraulic press at 241 kPa (35 psi) resulted in a slight increase in fvf and had the following make up:

Average Fiber Volume = 52.4%

Average Resin Volume = 46.2%

Average Void Volume = 1.4%

Average Infusion Time = 56 min

While there was success in increasing fvf, the decreased permeability of the preform resulted in an increase in infusion time of 26 min, which supports the experimentation previously mentioned (*36*).

One processing parameter that had not previously been optimized, for these materials, was the resin viscosity during infusion. In order to maintain the increased fvf and decrease the processing time, a second experimental process was conducted using a compacted and pre-heated preform (241 kPa [35 psi] at 48.9 °C [120 °F]). It was hypothesized that the heated preform would, in turn, heat the resin during infusion, thereby lowering the resin viscosity and infusion time. The resulting plates contained the following make up:

Average Fiber Volume = 56.6%

Average Resin Volume = 42.5%

Average Void Volume = 0.9%

Average Infusion Time = 49 min

This presented a 12.9% increase in fvf over our current VARTM process, allowed for a faster infusion time than that of the unheated preform, and resulted in a void content comparable to our control.

Next, experimentation was conducted to optimize the resin viscosity profile for the infusion process. The resin viscosity profile was analyzed using an Anton Parr MCR 501 rheometer with RheoPlus 32 software. Under ambient conditions, the viscosity of the resin system was determined to be approximately 375 cP (350 cP is the reported viscosity from the manufacturer), and that a significant decrease in viscosity (to below 110 cP) could be obtained by heating the resin system to 71 °C (160 °F). It was also determined that a large decrease in pot life (to less than 30 min) occurred after 51.7 °C (125 °F). Based on the time of infusion and the pot life of the resin system, it was determined that an optimal temperature of 48.9 °C (120 °F) would be used for the infusion. This resulted in a drop in the viscosity of the resin to approximately 120 cP. It was theorized that this decrease in viscosity of the resin would solve the issues with increased infusion times. The press-aided experimentation was recreated again, with temperature control of the resin added. The bulk resin was heated to 48.9 °C (120 °F), and held at this temperature for infusion. The press and preform were also heated to this temperature to maintain resin temperature during infusion and reduce potential gradient effect of the cured resin caused by uneven curing. The resulting thick cross-sectional composites had the following make up:

Average Fiber Volume = 60.4%

Average Resin Volume = 38.4%

Average Void Volume = 1.2%

Average Infusion Time = 32 min

This represents a 20.6% increase in fvf over our current double bag VARTM processing techniques for this material system. The average infusion time increase by 2 min over the control, but the overall processing time decreased by almost 12 h with the elimination of the long debulk under vacuum.

Fvf data were collected from at least three sample sets for each of the processing parameters discussed (processing parameters 2 and 3 had four sample sets). These data can be seen in figure 7. The low standard deviation suggest a high level of repeatability; however, further evaluation will be conducted in order to obtain a larger collection of sample sets.

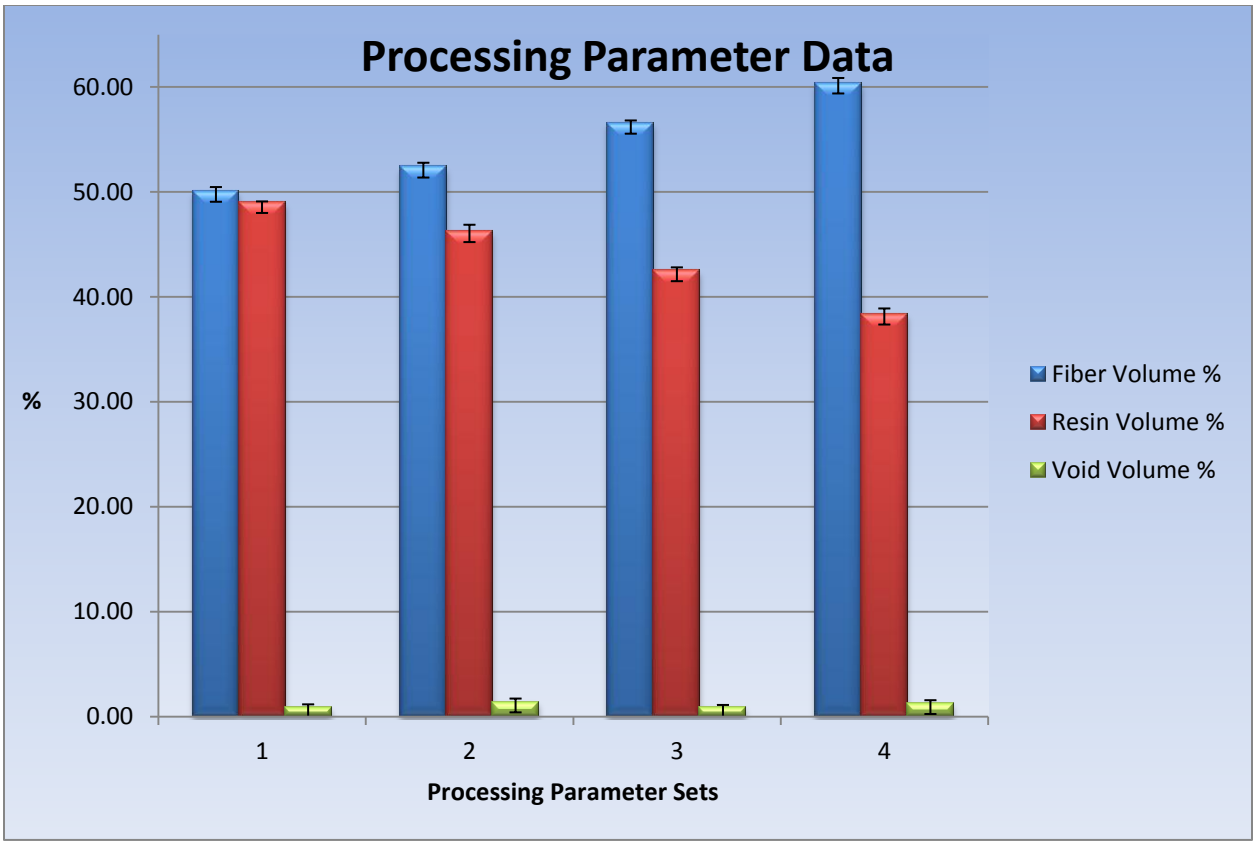

Figure 7. Percent content of experimental sets based on their respective processing parameters, including standard deviation.

4. Conclusions

A method for producing thick cross-sectional composites with a fvf percentage of above 60% has been shown. ARL's standard processing method for 813 g/m^2 (24 oz/yd^2) 5 x 5 plain weave S2 fiberglass with SC-15 epoxy resin allowed for the fabrication of thick cross-sectional composites

with an approximately 50/50 fiber-to-resin ratio. This is accomplished with an infusion time of 30 min for a 61 cm x 61 cm x 1.3 cm (24 in x 24 in x 0.5 in) laminate after a 12-h debulk cycle under 95 kPa (14 psi) vacuum. Using a hydraulic press, the 12-h debulk cycle is replaced with a 241 kPa (35 psi) compaction of the preform that is accomplished in less than 10 min. The press is heated, and, in turn, the preform is heated to a temperature allowing for viscosity control of the resin and optimal infusion. The resin is heated to the same temperature to ensure infusion time is completely optimized. For this system, the optimum infusion temperature was determined to be 48.9 °C (120 °F). Once compaction of the preform and infusion dynamics were optimized, the resulting thick cross-sectional composite laminates had an average fvf percentage of 60.4%. This represents a 20.6% increase in fvf over our current state-of-the-art processing techniques for this material system. The average infusion time increase by 2 min, as compared to the control, but the overall processing time decreased by almost 12 h with the elimination of the long debulk under vacuum.

Mechanical evaluations will follow in order to determine if forced nesting degrades or breaks the fiber, and will also be used to determine performance changes with higher fvf. Experimentation will continue in order to determine the maximum fvf attainable using this technique for this material system. The void content was increased from 0.9% in the control samples to 1.2% in the high fvf samples, and it will be determined if any negative impact results from this slight increase. Validation of experimentation will be conducted and tested for other material systems.

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List of Symbols, Abbreviations, and Acronyms

ARL	U.S. Army Research Laboratory
CAPRI	controlled atmospheric pressure resin infusion
fvf	fiber-volume fraction
MHI	Mitsubishi Heavy Industries
MRJ	Mitsubishi Regional Jet
SCRIMP	Seeman Composite Resin Infusion Molding Process
VAP	vacuum-assisted process
VARTM	vacuum-assisted resin transfer molding

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